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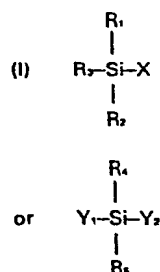
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(54) Lubricating agents for processing fibres and method of processing thermoplastic synthetic fibre filaments therewith.

(57) A lubricating agent for processing thermoplastic synthetic fibres contains at least one new type of silyl polyether obtainable by a reaction between polyether of a special type derivable by ring-opening addition polymerization of cyclic ether monomers and halogenated substituted silane. If such lubricating agent is used appropriately during the processing of thermoplastic synthetic fibres, both the coefficient of fibre friction and the rate of generating tar can be reduced. The silyl polyethers of use as shown by the formulae.



where R<sub>1</sub>-R<sub>4</sub> may be alike or different, each representing hydrogen, alkyl group, cycloalkyl group, allyl group, phenyl group, alkylphenyl group or benzyl group, R<sub>1</sub>-R<sub>3</sub> are not all hydrogen, R<sub>4</sub> and R<sub>4</sub> are not both hydrogen and X, Y<sub>1</sub> and Y<sub>2</sub> represent individually chlorine, bromine or iodine.

LUBRICATING AGENTS FOR PROCESSING FIBRES AND  
METHOD OF PROCESSING THERMOPLASTIC SYNTHETIC  
FIBRE FILAMENTS THEREWITH

Background of the Invention:

5           This invention relates generally to lubricating  
agents for use when processing fibres and to a method of  
processing fibre filaments by said lubricating agents;  
and more particularly to a novel type of lubricating  
agents for processing fibres which can both produce  
10   excellent lubrication and reduce the rate of tar  
generation and to a method of processing thermoplastic  
synthetic fibre filaments by using such lubricating  
agents.

          Fabrics are made of many kinds of thermoplastic  
15   synthetic fibres such as polyester, polyamide,  
polypropylene and polyacrylnitrile or cellulose-type  
fibres such as rayon, cupra and acetates as well as  
natural fibres. Many processes are involved in the  
fabrication such as weaving, drawing, false twisting,  
20   twisting and pasting although some of these processes  
may be combined into a single process. Various kinds of  
lubricating agents are used in these processes.

It has been well known that these lubricating agents must be able to produce excellent lubrication and to reduce the rate of tar generation. For this reason, use has been made not only of mineral oils and aliphatic esters but also of many kinds of lubricating agents having as main constituents, for example, polyoxyalkylene ethers (for example, U.S. Patent No. 3,338,830), esters of polyoxyalkylene ethers and aliphatic acids, formals of polyoxyalkylene alkyl ethers, esters of polyoxyalkylene bisphenols and aliphatic acids, orthosilicate esters and silicone alkylene oxide copolymers. Although they all have certain advantages, each of these lubricating agents for processing fibres is unsatisfactory in view of the aforementioned requirements. Mineral oils and aliphatic esters, for example, do not effectively inhibit the generation of tar. Polyoxyalkylene ethers and the aforementioned esters of bisphenol and aliphatic acid are not satisfactory in view of the lubricity requirement. Esters of polyoxyalkylene ethers and aliphatic acids, and the aforementioned formals tend to cause swelling in rubber-like substances. As for formals, furthermore, their yields by synthesis are low and there is also the problem of removing the formal-generating reagent. Orthosilicate esters have the disadvantage that hydrolysis can take place easily in aqueous solutions. As for silicone alkylene oxide copolymers, generation of tar on heating is not

sufficiently inhibited so that, for example a large amount of varnish-like tar is produced from polydimethyl siloxane.

It has thus been desirable to provide an improved  
5 lubricating agent for processing fibres which has improved properties when compared with the conventional lubricating agents. In order to improve production efficiency, on the other hand, attempts have also been made to increase the processing speed. Nowadays, high  
10 quality products are being introduced and fibre filaments are made smaller (lower denier) while there remains the problem that running filaments break, burrs appear on the filaments and heaters become covered with tar. For this reason, it is not merely desirable to  
15 make improvements on the conventional lubricating agents: there is a strong demand for new lubricating agents for processing fibres which can not only satisfy the requirements regarding yarn lubrication and generation of tar but also overcome the difficulties  
20 mentioned above.

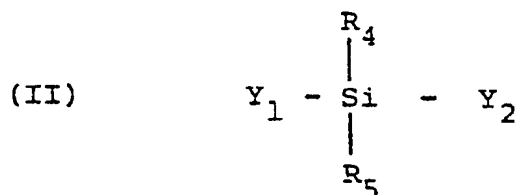
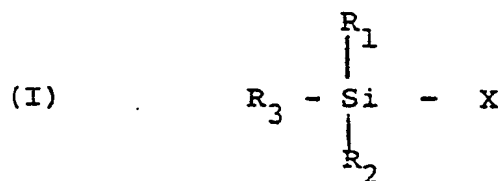
#### Summary of the Invention:

As a result of research for the development of a new lubricating agent for processing fibres which can satisfy these requirements, the present inventors have  
25 discovered that a lubricating agent for processing

fibres having as its main constituent a polyether  
 containing within its molecule a silicon atom combined  
 with certain specified groups is an appropriate choice  
 and that a superior result can be obtained if this  
 5 lubricating agent is appropriately applied to the fibre  
 filaments.

#### Detailed Description of the Invention

In one aspect, the present invention relates to a  
 lubricating agent for processing fibres, this  
 10 composition containing at least one silyl polyether  
 obtainable by a reaction between (a) a polyether which  
 is derived by a ring-opening addition polymerization of  
 cyclic ether monomers with 2 to 4 carbon atoms and  
 contains at least one hydroxyl group within its molecule  
 15 and (b) a halogenated substituted silane shown by one of  
 the following formulas (I) and (II):



where  $R_1$ - $R_5$  may be the same or different, each  
 representing hydrogen, alkyl group, cycloalkyl group,

allyl group, phenyl group, alkylphenyl group or benzyl group, while  $R_1$ - $R_3$  cannot all be hydrogen and  $R_4$  and  $R_5$  cannot both be hydrogen, and X,  $Y_1$  and  $Y_2$  are independently chlorine, bromine or iodine.

5           In another aspect, the present invention relates to a method of process thermoplastic synthetic fibre filaments according to which the filaments are lubricated by applying the aforementioned lubricating agent to the filaments at 0.1 to 3.0 weight percentage  
10           ratio during a step before the conclusion of the filament drawing and orientation.

          Silyl polyethers of the present invention are completely different in chemical structure from the conventional types of silicone alkylene oxide copolymers  
15           because they are obtained by mono-, di- or tri-substituted silylation of the end hydroxyl group of conventional types of polyether. They can be used as a stable aqueous solution or emulsion because they do not undergo hydrolysis easily. If they are applied to  
20           filaments, the coefficient of friction of the filaments can be reduced significantly. Another surprising effect which is obtained is that the amount of tar accumulated in heated machines (such as those for drawing and false twisting) can be reduced significantly. It is not  
25           clearly understood why the generation of tar can be so significantly reduced by this kind of silylation, but it

is thought that the terminal hydroxyl group of the polyether is implicated in the promotion of oxidizing thermal decomposition, and that its replacement by a silyl group has the effect of suppressing the generation of radicals as well as the chain transfer reaction.

The polyethers of the present invention can be classified into the following four groups from the point of view of their structures.

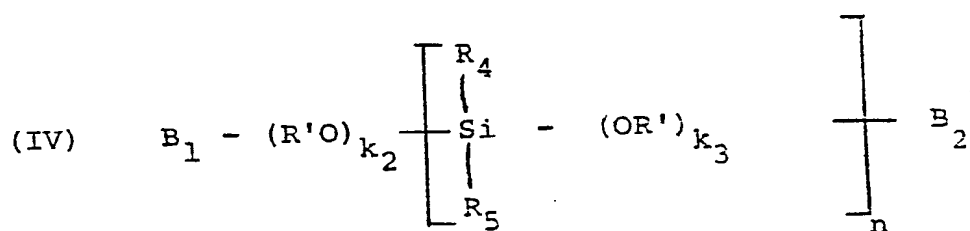
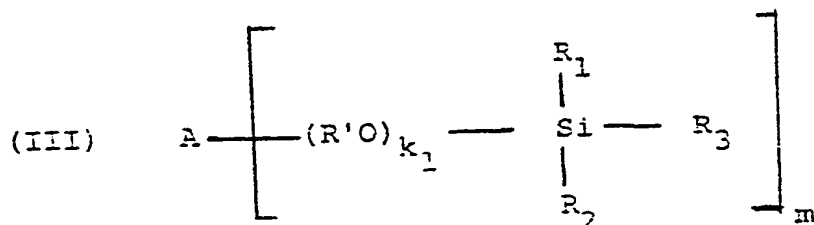
(1) The type of compounds obtainable by mono-, di- or tri-substituted silylation of the end OH group of a conventionally used type of polyoxyalkylene ether compound (such as the so-called non-ionic surface active agents of the polyoxyalkylene type, polyether, polyethylene glycol, polypropylene glycol, and polytetramethylene glycol). In this case, it is not necessary to silylate all OH groups inside the molecule.

(2) The type of compounds having the structure obtainable by the condensation of two molecules of polyoxyalkylene ether compounds of a conventional type having one OH group per molecule with one molecule of dichloro (dibromo or diiodo) mono- or di-substituted silane.

(3) The type of compounds having the structure obtainable by the polycondensation reaction of a

polyoxyalkylene ether of a conventional type having two OH groups per molecule and a dichloro (dibromo or diiodo) mono- or di-substituted silane and the type of compounds obtainable by plugging the end of a compound of the former type with a compound having one OH group or monochloro (monobromo or monoiodo) tri-substituted silane.

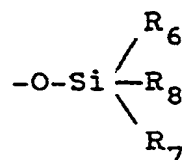
Of the above four groups, Groups (1)-(3) are superior from the point of view of lubricity. Silyl polyethers belonging to these groups can be described generally by the following two formulas:



where  $R_1-R_5$  are the same as in (I) and (II);  $R'$  may be the same or different among themselves, each representing an alkylene group with 2 to 4 carbon atoms;  $A$  represents monohydric to hexahydric alcohol (preferably with 1-18 carbon atoms), phenol, substituted phenol (preferably with 9-18 carbon atoms), carboxylic acid (preferably with 2 to 18 carbon atoms), alkyl



(preferably with 8 to 18 carbon atoms), or residual of  
 alkylene (preferably with 2 to 10 carbon atoms) - amine,  
 alkyl - or alkenyl (preferably with 2 to 18 carbon  
 atoms)- amide, thioether (preferably with 8 to 18 carbon  
 5 atoms) or mercaptan (preferably with 8 to 18 carbon  
 atoms);  $B_1$  and  $B_2$  each represent individually  
 hydroxyl group, alkoxy group, alkenoxy group, phenoxy  
 group, substituted phenoxy group (preferably with 9 to  
 18 carbon atoms), acyloxy group (preferably with 2 to 18  
 10 carbon atoms), alkyl- or alkenyl- amino (preferably with  
 8 to 18 carbon atoms) group, alkyl- or alkenyl- amide  
 (preferably with 2 to 18 carbon atoms) group or the  
 radical shown below:



15 where  $R_6-R_8$  are the same as in the case of  
 $R_1-R_5$ ;  $k_1-k_3$  are individually integers in the  
 range of 1 to 20 and may be the same or different;  $m$  is  
 an integer in the range of 1 to 6; and  $n$  is an integer  
 in the range of 1 to 10.

20 Silyl polyethers of the present invention, as shown  
 above, have various structures and molecular weights in  
 a wide range. Proper selection must be made of these,  
 depending on the type of fibres to which application is

to be made and the conditions under which these fibres are processed (such as the conditions of the heating processes). Fibres of the cellulose type, for example, have low fibre strength and since lubricity becomes an important factor for them, compounds with a relatively short polyoxyalkylene chain, or those with a low molecular weight (say, less than about 700), are preferable. Among thermoplastic synthetic fibres, filaments which are woven and knitted into flat yarns also are required excellent lubricity, so that those with a relatively low molecular weight (say, less than 700) are preferred. If the drawing temperature exceeds 200°C, however, those with a higher molecular weight are better suited for preventing fuming. For filaments which undergo a process of false twisting, those with molecular weight greater than about 700 are also preferable for preventing fuming. In the case of high-speed false twisting with yarn velocity speed in the range of 500 to 1000m/min, in particular, the lubricating agent tends to be scattered around by the centrifugal force of the rotary motion of the filament: those with molecular weight greater than about 1500 are preferable.

There will next be explained some examples of the synthesis of silyl polyethers of the present invention. The halogenated substituted silanes according to the aforementioned formulas (I) and (III), which are used

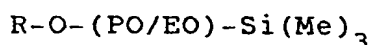
for the synthesis of such silyl polyethers, have 1 to 3 substituents and these substituents are alkyl groups (preferably with 1 to 18 carbon atoms), cycloalkyl groups (preferably an alkyl chain with 1 to 18 carbon atoms), allyl groups, phenyl groups, alkylphenyl groups (preferably an alkyl chain with 1 to 18 carbon atoms) or benzyl groups. They may be, for example, dimethylhydrogen chlorosilane, trimethyl chlorosilane, dimethyl dichlorosilane or diphenyl dichlorosilane. To commence, the polyether and a base such as pyridine are placed in a glass flask with a stirrer and a thermometer and the aforementioned halogenated substituted silane is added dropwise while the stirring is continued at a temperature below 40°C. Reaction is continued for 2 to 3 hours after the addition and the silyl ether is obtained by removing the by-product pyridine hydrohalides (hydrochlorides, hydrobromides or hydroiodides) after the end of the reaction.

Examples of polyethers to be used here include compounds obtained in the presence of a catalyst by block or random ring-opening addition polymerization of cyclic ether monomers such as ethylene oxide, propylene oxide, butylene oxide and tetrahydrofuran to alcohols such as methanol, ethanol, butanol, 2-ethylhexanol, dodecanol, stearyl alcohol, ethyleneglycol, glycerol, trimethylpropane, pentaerythritol, dipentaerythritol, etc; carboxylic acids such as capric acid, lauric acid,

adipic acid, sebacic acid, phthalic acid, trimellitic acid, pyromellitic acid, etc; amides of carboxylic acids such as lauric amide, oleic amide, stearic amide, etc.; amine-type compounds such as lauryl amine, oleyl amine, ethylene diamine, diethylene triamine, triethanol amine, etc; thioether-type or mercaptan-type compounds such as thioglycol, 1-thioglycerol, ethylene bis(2-hydroxyethyl) sulfide, triethyleneglycol dimercaptan, betaphenyl thioethanol, etc.

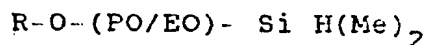
10        There are shown below individual examples of silyl polyethers thus synthesized and used according to the present invention but these examples are not intended to limit the scope of the present invention. In these examples the following symbols re used: Me for CH<sub>3</sub>; Ph  
15        for phenyl; Pe for phenylene.

(A)



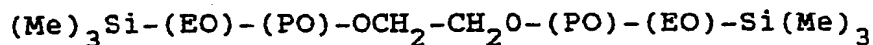
where R = C<sub>4</sub>H<sub>9</sub>; PO hereinafter indicates propyleneoxy group; EO hereinafter indicates  
20        ethyleneoxy group; PO/EO=50/50 (random coupling); and MW (average molecular weight) = 2000.

(B)



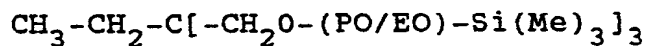
where R is a 1:1 mixture of  $C_{12}H_{25}$  and  $C_{13}H_{27}$ ;  
 PO/EO = 60/40; and MW = 2000.

(C)



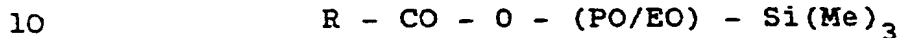
5 where PO/EO = 25/75 and MW = 2000.

(D)



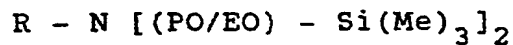
where PO/EO = 70/30 and MW = 2000.

(E)



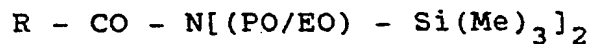
where R =  $C_{11}H_{23}$ ; PO/EO = 60/40; and MW = 2500

(F)



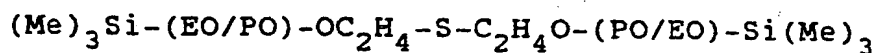
where R =  $C_{12}H_{25}$ ; PO/EO = 75/25; and MW = 2500.

15 (G)



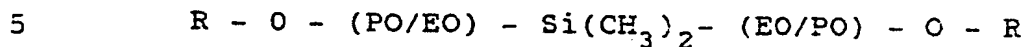
where R =  $C_{11}H_{23}$ ; PO/EO = 75/25; and MW = 2500.

(H)



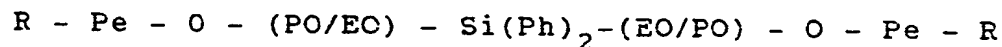
where PO/EO = 65/35 and MW = 2500.

(I)



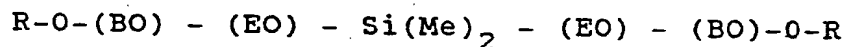
where R = CH<sub>3</sub>; PO/EO = 50/50; and MW = 2000.

(J)



where R = C<sub>4</sub>H<sub>9</sub>; PO/EO = 60/40; and MW = 2500.

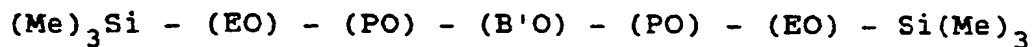
10 (K)



where R = C<sub>4</sub>H<sub>9</sub>; BO means 1,2-oxybutylene group;

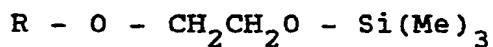
BO/EO = 30/70 (weight ratio); and MW = 2000.

(L)



where B'O means 1,4-oxybutylene group; B'O/PO/EO  
= 40/30/30 (weight ratio); and MW = 2000.

5 (M)

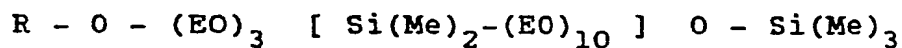


where R =  $\text{C}_{18}\text{H}_{35}$ .

(N)

10 where R =  $\text{C}_{12}\text{H}_{25}$ .

(P)



where R =  $\text{C}_{12}\text{H}_{25}$ .

There is no particular limitation regarding the  
15 concentration of these silyl polyethers in a lubricating  
agent of the present invention as long as the desired  
purposes of the present invention are achieved. The  
lubricating agents of the present invention,  
furthermore, may contain not only the silyl polyether

but appropriately also another lubricating agent, an antistatic agent, an emulsifier, a wetting agent, an anti-moulding agent and/or an anti-rusting agent.

Examples of lubricating agents that may be contained  
5 include refined mineral oils, aliphatic ether esters and polyethers derived from ethylene oxide or propylene oxide. A refined mineral oil with Redwood kinetic viscosity of 40 - 500 seconds at 30°C, for example, may be used. Among the esters of synthetic aliphatic acids,  
10 use may be made of esters of aliphatic monobasic acid and aliphatic monohydric alcohol, esters of polyhydric alcohol such as ethylene glycol, diethylene glycol, neopentyl glycol, trimethylol propane, glycerol, pentaerythritol, etc, and aliphatic monobasic acid or  
15 esters of aliphatic dibasic acid and aliphatic monohydric alcohol. Actual examples of esters of synthetic aliphatic acids include butyl stearate, n-octyl palmitate, 2-ethylehexyl palmitate, oleyl laurate, isohexadecyl laurate, isostearyl laurate,  
20 dioctyl sebacate, diisotridecyl adipate, ethylene glycol dioleate, trimethylol propane trioctanoate, pentaerythritol tetraoctanoate, etc. As for aliphatic ether esters, use may be made of ester of polyoxyethylene (5 mol) lauryl ether and lauric acid,  
25 diester of polyoxyethylene (5 mol) decyl ether and adipic acid, ester of polyoxyethylene (2 mol) polyoxypropylene (1 mol) octyl ether and palmitic acid, etc.



As for polyethers, use may be made of those obtainable by random or block addition polymerization of propylene oxide and ethylene oxide to methanol, ethanol, butanol, octanol, lauryl alcohol, stearyl alcohol, etc., those  
5 obtainable by random or block addition polymerization of propylene oxide and ethylene oxide to polyhydric alcohol such as propylene glycol, trimethylol propane, glycerol, pentaerythritol, sorbitol, etc. with molecular weights in a wide range.

10 Examples of aforementioned antistatic agents include anionic surface active agents such as sulfonates, phosphates and carboxylates, cationic surface active agents of the quaternary ammonium salt type and amphoteric surface active agents of the imidazoline  
15 type, betaine type and sulfobetaine type, while examples of aforementioned non-ionic surface active agents include polyoxyethylene alkyl ethers, polyoxyethylene alkylphenyl ethers, polyoxyethylene alkyl esters and partial alkyl esters of polyhydric alcohols.

20 The lubricating agents of the present invention show their effectiveness when applied to fibres as spin finish or as coning oil. They may be applied to fibres either as an aqueous emulsion, a solution with an organic solvent or by themselves (straight oiling). The  
25 amount of lubricating agent deposited on the fibre is usually 0.20-2.0 weight % when applied as spin finish

lubricant and 0.5-3.0 weight % when applied as coning oil.

The lubricating agents of the present invention explained above exhibit high levels of effectiveness when they are applied to thermoplastic synthetic fibres such as polyesters, polyamides, polypropylene, polyacrylonitrile, etc., cellulose-type fibres such as rayon, cupra, acetates, etc, and also many types of natural fibres. A comparison with the conventional lubricating agents and their constituents show that aforementioned silyl polyethers which play central roles in the lubricating agents of the present invention bring about superior lubricating capability and ability to reduce generation of tar. Moreover, these silyl polyethers have many advantages regarding their production such that they can be synthesized easily and that compounds which did not participate in the reaction can be removed easily.

When they are used in the production process of thermoplastic synthetic fibres such as polyesters, polyamides, polypropylene and polyacrylonitrile, they are particularly effective if they are applied at the rate of 0.1-3.0 weight % or preferably 0.2-2.0 weight % with respect to such thermoplastic synthetic fibres and also if the application is made during a step prior to the completion of the drawing and orientation of the

fibres, because the aforementioned effects can continue throughout the subsequent production processes (inclusive of heating processes).

In order to explain the present invention more concretely, there will subsequently be shown examples of synthesis of silyl polyethers (hereinafter abbreviated as Si-PE) according to the present invention together with evaluation of their characteristics. In what follows, symbols Si-PE(A)-(P) will refer to the individual examples (A)-(P) of silyl polyethers illustrated before.

Example of synthesis No. 1 (synthesis of Si-PE(A)):

Polyether of MW = 2000 (500g, or 0.25 mol) obtained by random addition polymerization with PO and EO in weight ratio of 50:50 and n-butanol was placed in a glass reaction vessel of volume 1 litre (with an agitator and a reflux condenser) and after 22.75g (0.25 mol) of pyridine was added and stirred to make a uniform mixture, 27.125g (0.25 mol) of trimethyl chlorosilane was gradually added from a dropping funnel at a reaction temperature below 40°C. The temperature was maintained below 40°C even after the addition was completed and the reaction was continued for 2 to 3 hours. Pyridine hydrochloride separates as the reaction goes on. The system pressure was reduced after the completion of the

reaction, and after the temperature was raised to about 100°C and small amounts of unreacted pyridine and trimethyl chlorosilane were removed from the system, the pyridine hydrochloride was removed and the reaction  
5 product (silyl polyether) was obtained.

According to an analysis by the proton nuclear magnetic resonance method (hereinafter abbreviated as NMR), the reaction ratio (fraction of the OH Group of polyether converted into trimethylsilyl group) was about  
10 80%.

Example of synthesis No. 2 (synthesis of Si-Pe(I)):

Polyether of MW = 1000 (500g, or 0.5 mol) obtained by random addition polymerization with PO and EO in weight ratio of 50:50 and methanol was mixed with 45.5g  
15 (0.5 mol) of pyridine and 32.25g (0.25 mol) of dimethyl dichlorosilane and reaction product was obtained by using the same apparatus and method of operation as in the previous example. The reaction ratio was about 90% by an NMR analysis.

20 Examples of test and comparison experiments Nos. 1-5:

Lubricating agents for test and comparison experiments Nos. 1-5 shown in Table 1 were individually prepared. A 10-weight % emulsion each of these

lubricating compositions was applied individually by the  
kiss-roll method onto commercially available nylon  
filaments (semi-dull 70-denier 24-filament) which had  
been degreased with cyclohexane and dried. The amount  
5 of lubricant deposited was 0.8-1.0 weight % on fibre.  
The coefficient of friction was measured for each  
filament and the rate of tar generation was measured for  
each lubricating composition. The results are shown in  
Table 1 wherein examples of test and comparison  
10 experiments assigned the same number correspond to each  
other, showing the silylation effects on polyether. One  
can see from the results of Table 1 that the lubricating  
agents of the present invention have lower coefficients  
of friction and lower rates of tar generation than those  
15 of conventional types.

Evaluation of the characteristics described in Table  
1 was made in the following ways:

(i) Measurement of coefficient of friction

A lubricated nylon filament was used for measurement  
20 by a  $\mu$ -meter (made by Eiko Sokuki Kabushiki Kaisha)  
under the following conditions: friction pin =  
cylindrical plated pin of 25 mm in diameter; contact  
angle between filament and friction pin = 90°; initial  
tension ( $T_1$ ) = 20g; sliding speed of filaments =  
25 300m/min; environment = 25°C x 65% RH. The filament

tension immediately after the friction pin was measured ( $T_2$ ) and the coefficient of friction was calculated by the following formula:

$$\text{Coefficient of friction} = A \ln T_2/T_1$$

5 where A is a factor determined by the angle of contact and ln is the natural logarithm. Lubricity is the better, the smaller the coefficient of friction.

(ii) Measurement of the rate of tar generation

Three grams of lubricating agent was weighed  
10 accurately in a stainless steel dish having diameter of 8cm and depth of 8mm. The dish was then placed in a heater box at the temperature of 230°C for 48 hours and cooled in a dessicator, and was again weighed accurately. The ratio of the tarry residue to the  
15 effective content of original lubricant was calculated from the data obtained before.

## (iii) Standards of evaluation

Coefficient of friction		Rate of tar generation	
0	Less than 0.03	0	Less than 10%
+	0.30 - 0.35	+	10 - 20%
++	0.35 or over	++	20% or over

5

TABLE 1

		Test					Comparison				
		1	2	3	4	5	1	2	3	4	5
5	Si-PE(E)	50									
	Si-PE(F)		50								
	Si-PE(G)			50							
	Si-PE(H)				50						
	Si-PE(J)					50					
10	(E1)						50				
	(F1)							50			
	(G1)								50		
	(H1)									50	
	(J1)										50
15	*1	20	20	20	20	20	20	20	20	20	20
	*2	25	25	25	25	25	25	25	25	25	25
	*3	5	5	5	5	5	5	5	5	5	5
20	Coef.of Friction	0	0	0	0	0	+	+	+	++	++
	Tar gener- ation	0	0	0	+	+	+	+	+	++	



In Table 1, the numbers are in units of weight %.

\*1 = octyl stearate, \*2 = (POE(8)lauryl ether,

\*3 = potassium dodecanyl succinate,, and

(E1):  $R - \text{COO} - (\text{PO/EO}) - \text{H}$

5 where  $R = \text{C}_{11}\text{H}_{22}$ , PO/EO = 60/40 and MW = 2500;

(F1):  $R - \text{N} [(\text{PL/EO}) - \text{H}]_2$

where  $R = \text{C}_{12}\text{H}_{25}$ , PO/EO = 75/25 and MW = 2500;

(G1):  $R - \text{CON} [(\text{PO/EO}) - \text{H}]_2$

where  $R = \text{C}_{11}\text{H}_{23}$ , PO/EO = 75/25 and MW = 2500;

10 (H1):  $\text{S} [\text{C}_2\text{H}_4 - \text{O} - (\text{PO/EO}) - \text{H}]_2$

where PO/EO = 65/35 and MW = 2500;

(J1):  $R - \text{Pe} - \text{O} - (\text{PO/EO}) - \text{CH}_2 - (\text{EO/PO}) - \text{O} - \text{Pe} - \text{R}$

where  $R = \text{C}_4\text{H}_9$ , PO/EO = 60/40 and MW = 2500.

Examples of test and comparison experiments Nos. 6-11:

15 The lubricating agents for test and comparison experiments Nos. 6-11 shown in Table 2 were individually prepared. A 10% weight of emulsion each of these lubricating agents was applied individually by kiss-roll method onto commercially available polyester filaments

(semi-dull 75-denier 36-filament) which had been degreased by cyclohexane and dried. The amount of lubricant deposited on the fibre was 0.4-0.6 weight %. Coefficient of friction and the rate of tar generation were measured as before. The results are shown in Table 2 wherein examples of test and comparison experiments assigned the same number correspond to each other, showing the silylation effects on polyether. One can see also from the results of Table 2 that the lubricating agents of the present invention have lower coefficients of friction and lower rates of tar generation than those of conventional types.

(i) Standards of evaluation

Coefficient of friction		Tar generation	
O	0.30-035	O	less than 1%
+	Over 0.35	+	1 - 5%

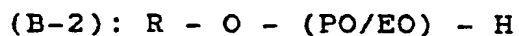
TABLE 2

		Test						Comparison					
		6	7	8	9	10	11	6	7	8	9	10	11
5	Si-PE(A)	98											
	Si-PE(B)		98										
	Si-PE(C)			98									
	Si-PE(D)				98								
	Si-PE(I)					98							
	Si-PE(K)						98						
10	(A-2)							98					
	(B-2)								98				
	(C-2)									98			
	(D-2)										98		
	(I-2)											98	
	(K-2)												98
15	*4	2	2	2	2	2	2	2	2	2	2	2	2
	Coef.of Friction	0	0	0	0	0	0	0	+	+	+	+	+
20	Tar generation	0	0	0	0	0	0	0	+	+	+	+	+

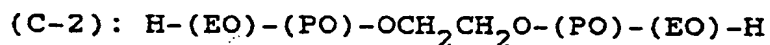
In Table 2, \*4 is sodium alkylsulfonate and

(A-2): R - O - (PO/EO) - H

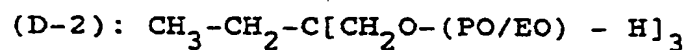
where R = C<sub>4</sub>H<sub>9</sub>, PO/EO = 50/50 and MW = 2000;



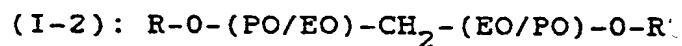
where R = 1:1 mixture of  $C_{12}H_{25}$  and  $C_{13}H_{27}$ ,  
PO/EO = 60/40 and MW = 2000;



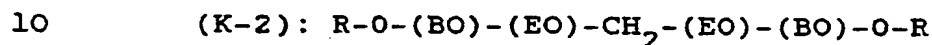
5 where PO/EO = 25/75 and MW = 2000;



where PO/EO = 70/30 and MW = 2000;



where R =  $CH_3$ , [P/EO = 50/50 and MW = 2000; and



where R =  $C_4H_9$ , BO/EO = 30/70 and MW = 2000.

Examples of test and comparison experiments Nos.12 and  
13:

The lubricating agents for test and comparison  
15 experiments Nos. 12 and 13 shown in Table 3 were  
individually prepared. Each of these lubricating agents

was applied by the neat oiling method to commercially available acetate filaments (bright 75-denier 20-filaments) degreased by diethyl ether. The amount of lubricant deposited on the fibre was 1.5-2.0 weight %.

5 Coefficient of friction was measured as before in the case of Table 1 and evaluated according to the following standards. The results are shown in Table 3. One can see also from the results of Table 3 that the lubricating agents of the present invention have lower

10 coefficients of friction than the mineral oils which have been used conventionally as smoothening agent for lubricants for acetates.

Standards of evaluation:

15

Coefficient of friction	
OO	less than 0.22
O	0.22 - 0.25
+	0.25 - 0.28
++	0.28 or greater

Table 3

	Test		Comparison	
	12	13	12	13
Si-PE(M)	90			
Si-PE(N)		90		
Mineral oil (60RW second)			90	
Mineral oil (90RW second)				90
Sorbitan monooleate	10	10	10	10
Coefficient of friction	00	0	+	++

10 Examples of test experiments Nos. 14-17 and comparison  
experiments Nos. 14-16

15 The lubricating agents for test experiments Nos. 14-17 and comparison experiments Nos. 14-16 shown in Table 4 were individually prepared. A partially oriented yarn (POY) was prepared for each case by the method described below and such POY was used for draw - false twist - texturing and studies were made about the following items: (1) cross yarn of POY, (2) friction coefficient of POY, (3) appearance of fuzz on

drawtexturing yarn, and (4) the amount of tar on the heaters. The results are shown in Table 4. One can see from the results of Table 4 that the POY cross yarn, the tar generation, fuzz of draw textured yarn and the  
5 coefficient of friction are small if a lubricating agent of the present invention is used.

#### Production of POY

Immediately after melt spinning of polyethylene terephthalate, a 10%-emulsion of lubricating agent was  
10 applied by the kiss-roll method and a 12-kg cake of POY with 115 denier 36 filaments was obtained by winding at the rate of 3500m/min. The amount of lubricant deposited on POY was 0.4-0.5 weight %.

#### (ii) Draw - false twist texturing

15 Twisting system = three-axis friction method (hard urethane rubber disk); Speed of yarn = 600m/min; Draw ratio = 1.518; Heater on twist side = 2m in length with surface temperature of 220°C; Heater on untwisting side = none;

20 Intended number of turns = 3200T/m.

#### (iii) Evaluation of cross yarn on POY cake

It was examined by observation whether any filament was slipping off in a straight line on the side surface

of the POY cake. Its occurrence can cause the filament to break when a POY is unwound in a draw texturing process.

(iv) Evaluation of friction coefficient of POY

- 5           Coefficient of friction was measured in the same way as for Table 1 except that polyester POY was used for testing. Evaluation was made by the following standards:

O = Coefficient of friction smaller than 0.35  
+ = Coefficient of friction 0.35 or greater

10          (v) Evaluation of appearance of fuzz

It was examined by observation whether there was fuzz generated on the side surface of the cheese (2-kg roll) of false twisted yarn.

(vi) Evaluation of tar on heaters

- 15           After a continuous operation for 10 days under the aforementioned conditions of draw-false twisting, a magnifier was used to examine by observation whether or not tar had been generated in the filament passage on the surface of heater. Evaluation was made by the  
20          following standards:



O = Substantially no tar adhesion observed

+ = Some tar adhesion observed

Table 4

5		Test				Comparison		
		14	15	16	17	14	15	16
	Si-PE(A)		60	30				
	Si-PE(I)	99						
	Si-PE(P)				30			
	(A-2)						60	30
10	(I-2)		39	69	69	99	39	69
	Sodium alkylsulfonate	1	1	1	1	1	1	1
	Cross Yarn on POY Cake	No	No	No	No	No	No	No
	Coef. of POY friction	O	O	O	O	+	+	+
	Fuzz on POY Cake	No	No	No	No	No	No	No
15	Tar on heaters	O	O	O	O	+	+	+

In Table 4, (A-2) and (I-2) are as defined for Table 2.

Examples of test experiments Nos. 18 and 19

5 Polyester POY was prepared by using a lubricating agent having the following composition and by the same method used for Table 4 (Experiment No.18):

	Si-PE(K)	45 weight%
	Isooctyl palmitate	10 weight%
	$C_{12}H_{25}O[(C_3H_6O)_{20}(C_2H_4O)_{18}]_B H$	40 weight%
10	Sodium alkylsulfonate	5 weight%

where B represent block polymerization structure.

This POY was draw-false twisted by the same method as before for Table 4 and 1.5-2.0% weight of a lubricating agent of the following composition was applied (Experiment No.19) as coning oil immediately before the fibre was wound up:

	Si-PE(M)	60 weight %
	Mineral oil (60-second Redwood)	30 weight %
	Sorbitan monooleate	5 weight %
20	POE (5 mol) nonylphenyl ether	4 weight %
	10 cst/30°C dimethylsilicone	1 weight %

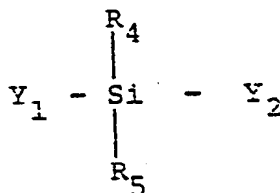
This false twisted yarn was used for weaving with a water jet loom but no problem was observed at all and good results were obtained.

## CLAIMS:

1. A lubricating agent for processing fibres comprising at least one kind of silyl polyether obtainable as a reaction product between polyether  
 5 which is derived by ring-opening addition polymerization of cyclic ether monomers with 2 to 4 carbon atoms and the molecule of which contains at least one hydroxyl group and halogenated substituted silane shown by formulae

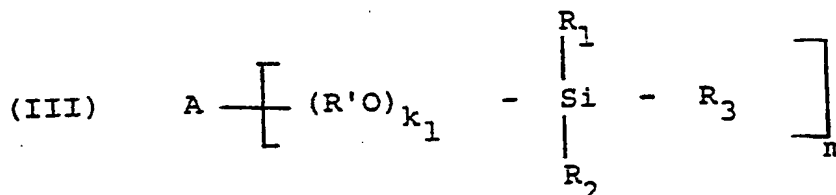


or

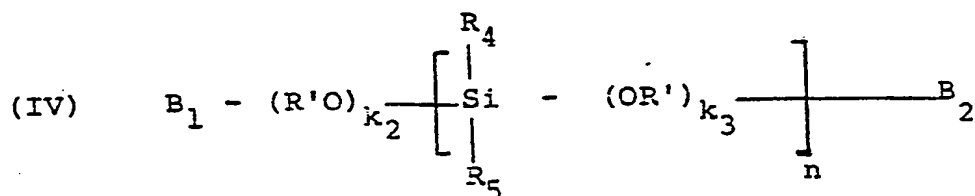


- where  $R_1-R_5$  may be alike or different, each representing hydrogen, alkyl group, cycloalkyl group,  
 15 allyl group, phenyl group, alkylphenyl group or benzyl group.  $R_1-R_3$  are not all hydrogen,  $R_4$  and  $R_5$  are not both hydrogen and X.  $Y_1$  and  $Y_2$  represent individually chlorine, bromine or iodine.

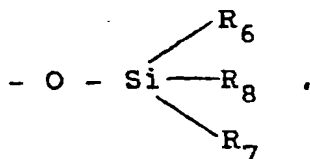
2. The lubricating agent of claim 1 wherein said silyl  
 20 polyether is a compound shown by formula



or



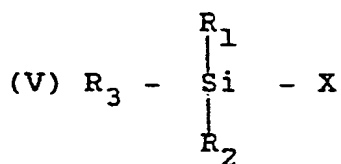
where R' are similar or different, each representing  
 5 alkylene group with 2 to 4 carbon atoms; A is  
 monohydric-hexahydric alcohol, phenol, substituted  
 phenol, carboxylic acid, alkyl or alkenyl or residual  
 of alkenyl or alkylene amine, alkyl or alkenyl, amide,  
 thioether or mercaptan; B<sub>1</sub> and B<sub>2</sub> represent  
 10 individually hydroxyl group, alkoxy group, alkenoxy  
 group, phenoxy group, substituted phenoxy group,  
 acyloxy group, alkyl or alkenyl amino group, alkyl or  
 alkenyl amide group or



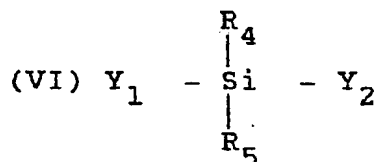
15 R<sub>6</sub>-R<sub>8</sub> being defined similarly as R<sub>1</sub>-R<sub>5</sub> for (I)  
 and (II); k<sub>1</sub>-k<sub>3</sub> are individually an integer in the  
 range of 1-200 and may be alike or different; m is an  
 integer in the range of 1-6; and n is an integer in

the range of 1 - 10.

3. A method of processing a thermoplastic synthetic fibre filaments comprising the step of lubricating said fibre filaments by applying thereonto a lubricating agent at a rate of 0.1-3.0 weight % with respect to said fibre filaments, said lubricating agent comprising at least one kind of silyl polyether obtainable by a reaction between polyether having one or more hydroxyl group in its molecule and derivable by ring-opening addition polymerization of cyclic ether monomers with 2 to 4 carbon atoms and halogenated substituted silane shown by formula



or



where  $R_1$ - $R_5$  may be alike or different, each representing hydrogen, alkyl group, cycloalkyl group, allyl group, phenyl group, alkylphenyl group or benzyl group.  $R_1$ - $R_3$  are not all hydrogen.  $R_4$  and  $R_5$

are not both hydrogen, and X, Y<sub>1</sub> and Y<sub>2</sub> represent individually chlorine, bromine or iodine.

4. The method of claim 3 further comprising the step of drawing and orienting said filaments, said
- 5 lubricating step taking place prior to said step of drawing and orienting.

(12)

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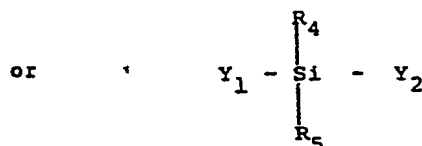
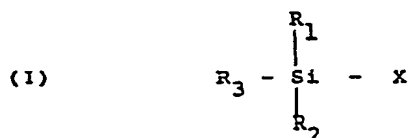
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(54) **Lubricating agents for processing fibres and method of processing thermoplastic synthetic fibre filaments therewith.**

(57) A lubricating agent for processing thermoplastic synthetic fibres contains at least one new type of silyl polyether obtainable by a reaction between polyether of a special type derivable by ring-opening addition polymerization of cyclic ether monomers and halogenated substituted silane. If such lubricating agent is used appropriately during the processing of thermoplastic synthetic fibres, both the coefficient of fibre friction and the rate of generating tar can be reduced. The silyl polyethers of use as shown by the formulae.

where R<sub>1</sub>-R<sub>5</sub> may be alike or different, each representing hydrogen, alkyl group, cycloalkyl group, allyl group, phenyl group, alkylphenyl group or benzyl group, R<sub>1</sub>-R<sub>3</sub> are not all hydrogen, R<sub>4</sub> and R<sub>5</sub> are not both hydrogen and X, Y<sub>1</sub> and Y<sub>2</sub> represent individually chlorine, bromine or iodine.







European Patent  
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## EUROPEAN SEARCH REPORT

0132910

Application number

DOCUMENTS CONSIDERED TO BE RELEVANT			EP 84302050.4
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.) 3
A	<u>GB - A - 2 053 942</u> (OLIN) * Abstract *	1,2	D 06 M 15/647 C 08 L 71/02 C 08 L 83/00
A	PATENT ABSTRACTS OF JAPAN, unexamined applications, C field, vol. 5, no. 152, September 25, 1981 THE PATENT OFFICE JAPANESE GOVERNMENT page 145 C 73 * Kokai-no. 56-84 751 (IDEMITSU) *	1	
D,A	<u>US - A - 3 338 830</u> (STOKES)		
			TECHNICAL FIELDS SEARCHED (Int. Cl.) 3
			D 06 M C 08 L C 08 G
The present search report has been drawn up for all claims			
Place of search VIENNA		Date of completion of the search 22-10-1985	Examiner FARNIOK
CATEGORY OF CITED DOCUMENTS			
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	

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